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SOME PHYSICAL CONSTANTS OF A FEW HYDROCARBONS AND THEIR STRUCTURAL ISOMERS

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1. Introduction

In previous papers it has been shown that in the homologous series of normal saturated hydrocarbons [22],3 normal aliphatic alcohols [23], and acetates of normal aliphatic alcohols [24], when containing more than some small number, n, of carbon atoms (in the case of acetates beginning with the first member) the addition of a CH2 group to the normal alkyl group to form the next higher normal alkyl group results in an increase of approximately 0.0029° C. per mm. of mercury in the coefficient dt/dp at the normal boiling point. Since this value was found to be independent of the chemical nature of the compound, the additivity of the effect of the CH2 group on dt/dp was postulated. However, n-nonane, which has a boiling point of 150.81° C. and a dt/dp of 0.0497° C. per mm. of mercury, has been found to deviate somewhat from this rule. It is, then, important to examine isomeric compounds for further tests of the postulate.

This paper describes measurements of dt/dp coefficients of five isomeric hexanes: n-hexane,

2-methylpentane, 3-methylpentane, 2, 3-dimethylbutane, and 2, 2-dimethylbutane; of six isomeric heptanes: n-heptane, 2, 3-dimethylpentane, 2, 2-dimethylpentane, 3-ethylpentane, 3-methylhexane, and 2, 2, 3-trimethylbutane; of two isomeric octanes: n-octane and 2, 2, 4-trimethylpentane; of two isomeric nonanes: n-nonane and 2, 5-dimethylheptane; and of the three isomeric xylenes. In addition, there are included for comparison the results of previous measurements on the isomeric nonanes: 2-methyloctane, 3-methyloctane, 4-methyloctane and 2, 6-dimethylheptane. There are also reported the boiling points of these compounds and the freezing points of the isomeric hexanes.

2. Methods of Measurements

For determining boiling points, W. Swietos-lawski's ebulliometric technic [15], a comparative method with water as a primary reference standard, was used. Details of the experimental procedure for measurements of boiling point and dt/dp have been reported briefly in former papers [22, 2].

For calculating the normal boiling point, the formula [22]

$$t_s = t_s' + \frac{\Delta t_s}{\Delta t_w} (100 - t_w') \tag{1}$$

was used. In this equation t_s represents the normal boiling point of the substance under investigation, t_s' the boiling point of water actually measured under the same pressure as that of the substance. The ratio, $\Delta t_s/\Delta t_w$, of the change in boiling point of the substance to the change in boiling point of water at normal pressure was computed from the boiling point of the substance under investigation and that of water under the same pressure, slightly above and slightly below 1 atm.

By a method which has been described previously [22, 25], the boiling points measured were corrected whenever possible by extrapolation, to minimize the effect of impurities in the preparations investigated.

 $^{^{\}rm 1}\,{\rm Publication}$ approved by the Director of the National Bureau of Standards of the Department of Commerce.

² Guest worker at the National Bureau of Standards from the Polytechnic Institute, Warsaw, Poland.

³ Numbers in brackets throughout the text indicate references listed at the end of the paper.

All temperature measurements were made with a platinum resistance thermometer and Mueller bridge, calibrated at this Bureau.

3. Materials Used

The isomeric hexanes used were synthetic materials kindly furnished by L. Cramer and M. J. Mulligan [6] of the General Motors Corporation. Each of the substances investigated was examined for purity in a Swietoslawski standard differential ebulliometer [15]. The ebulliometric test, which is based on measuring the difference Δt between the boiling point and temperature of condensation of the substance, indicated high purity, all of the hexanes being of the fifth or of the fourth degree of purity on Swietoslawski's scale.

Isomeric heptanes were obtained from G. Calingaert of the Ethyl Gasoline Corporation. These preparations, although not of the highest purity, were satisfactory for measurements of dt/dp, since these constants are not very sensitive to small quantities of impurities. The sample of 2, 2-dimethylpentane, kindly furnished by F. W. Rose, Jr., of the American Petroleum Institute Research Project 6 at this Bureau, was a preparation of the fifth, i. e. the highest degree of purity.

The *n*-nonane was obtained from S. T. Schicktanz and the isomeric xylenes from J. D. White and F. W. Rose, Jr., research associates of the American Petroleum Institute. The values given for 2, 2, 4-trimethylpentane are those reported recently by E. R. Smith and H. Matheson [14].

4. Experimental Results and Conclusions

(a) Isomeric hexanes.—The data obtained for the isomeric hexanes are given in Table 1, which shows for each substance the difference, Δt , between boiling point and condensation temperature, the normal boiling point, the value of dt_s/dt_w (the ratio of the change of boiling point of each substance to the change of the boiling point of water, caused by a small increase or decrease of pressure, obtained directly in these comparative measurements), and the value of dt/dp (the increment of boiling point of the substance under investigation per millimeter change of pressure at 1 atm.). The value for n-hexane was calculated from the dt_s/dt_w ratio given in a former paper [22], taking the value 0.036858° C. per mm. of mercury for the dt/dp coefficient of water reported by Beattie and Blaisdell [1]. This figure was used

for computing the dt/dp coefficients of all the hydrocarbons investigated.

The data for dt_s/dt_w permit the calculation of the correction for normal boiling points of the substances from equation 1, within the pressures expressed by the boiling points reported for water, or even when extrapolated for somewhat larger pressure differences. In the latter case, the accuracy of the results decreases with increase in magnitude of the pressure difference.

The data for dt_s/dt_w may also be used to compute the value of the dt/dp coefficient corresponding to any pressure within the reported limits by the use of the equation [15]

$$\left(\frac{dt}{dp}\right)_{s} = \frac{dt_{s}}{dt_{w}} \left(\frac{dt}{dp}\right)_{w}$$

The purity of the isomeric hexanes used was confirmed by examining the freezing curves, using an apparatus similar to that of Mair [11].

The freezing curve of the 2-methylpentane was practically flat. A rise in the freezing curve was observed at the first stage of freezing, when the liquid was seeded, and a slight decrease was noticed at the end of the measurement, when the mixture of liquid with crystals was so viscous that stirring of the material was almost impossi-Obviously, the heat exchange under the conditions at the end of the measurement was not adequate to maintain thermal equilibrium in the system. The shape of the freezing curve indicated high purity of the substance. than two-thirds of the sample crystallized at constant temperature within ± 0.003° C. This is in agreement with the ebulliometric test of Indeed the discrepancy between the boiling point and the condensation temperature for the given sample of 2-methylpentane was found to be only 0.002° C., which also classifies the preparation as very pure.

The flatness of a freezing curve proves that in the substance investigated there are only slight amounts of impurities which might be removed by fractional crystallization. The small difference between the boiling point and condensation temperature, as measured in the differential ebulliometer, shows that there are but slight amounts of impurities that might be removed by distillation. Since there is only a very small probability that there can be impurities present having both boiling and freezing points almost equal to those of the substances investigated, the claim of high purity for 2-methylpentane is sub-

TABLE 1 Data for Isomeric Hexanes

Substance	Δt	Normal boiling point	dt_s/dt_w	Boiling point of water	$(dt/dp)_{76}$
	° C	° C		° C	° C/mm
2-Methylpentane	0.002	60.267	1.1261	100.888	0.0414
			1.1236	100.000	
			1.1182	98.022	
3-Methylpentane	.005	63.256	1.1374	100.961	.0418
			1.1354	100.000	
			1.1319	98.334	
2, 3-Dimethylbutane	.004	58.022	1.1346	101.091	.0417
			1.1311	100.000	
			1.1286	98.428	
2, 2-Dimethylbutane	.009	49.804	1.1188	101.137	.0411
			1.1163	100.000	
			1.1129	98.503	
n-Hexane	.005	68.733	1.1383	101.075	.0419
			1.1363	100.000	
			1.1324	97.948	
				Average	0.0416

TABLE 2 Comparison of Data for Isomeric Hexanes

Substance	Author	Normal Boiling point	Freezing point	$(dt/dp)_{760}$
		° C	° C	° C/mm
2-Methylpentane	Chavanne [5]	60.2		_
	Maman [12]	60.5	_	_
	Bruun and Hicks-Bruun [4]	60.4	-143 ± 0.5	
	Edgar and Calingaert [8]	60.2		_
	Cramer and Mulligan [6]	60.22 to 60.26	_	_
	Wojciechowski	60.26_{7}	-153.7_{1}	0.0414
3-Methylpentane	Chavanne [5]	63.2	_	-
	Maman [12]	62.7	_	
	Bruun and Hicks-Bruun [4]	63.3	-118 ± 0.5	
	Edgar and Calingaert [8]	63.2	_	_
	Cramer and Mulligan [6]	63.16 to 63.21	_	_
	Wojciechowski	63.256	-	0.0418
2, 2-Dimethylbutane	Chavanne [5]	49.7		
	Maman [12]	52.9	_	
	Cramer and Mulligan [6]	49.80 to 49.82	-	_
	Wojciechowski	49.804	-102	.0411
2, 3-Dimethylbutane	Chavanne [5]	58.1	-	_
	Maman [12]	58.2		_
	Bruun and Hicks-Bruun [4]	58.0	-135 ± 1	_
	Edgar and Calingaert [8]	58.1	_	
	Cramer and Mulligan [6]	57.82 to 58.02	_	-
	Wojciechowski	58.022	-132.7	.0417

stantiated, and the value -153.7_1° C. for its freezing point seems reliable.

The freezing point of 3-methylpentane could not be measured because the substance supercooled without crystallization and became so viscous that stirring was impossible. All attempts failed to cause crystallization by seeding or by local extreme supercooling.

2, 3-Dimethylbutane and 2, 2-dimethylbutane were also very difficult to crystallize by seeding. For this reason the freezing points of these two compounds, $-132.7 \pm 1^{\circ}$ C. and $-102.0 \pm 1^{\circ}$ C., respectively, can not be reported with better accuracy than 1° C.

Taking into consideration the high purity of the substances, with the exception of 2, 2-dimethylbutane, it is believed that the data obtained for the boiling points can hardly differ by more than 0.01° from the true boiling points of these compounds. The values for dt/dp can be affected only very slightly by the presence of small amounts of impurities, and it is possible that the data for 2, 2-dimethylbutane alone might be significantly in error for this reason.

The data contained in Table 1 show that the values of the coefficient dt/dp for all the hexanes fluctuate, probably because of the rearrangements in the chain, within about 1 per cent of the average value of 0.0416° C/mm.

In Table 2 the author's data for isomeric hexanes are compared with values previously reported.

(b) Isomeric heptanes.—The data for the five isomeric heptanes are reported in Table 3, where the headings are self-explanatory.

The boiling points contained in Table 3 are the actual data of measurements carried out within a precision of 0.001° C. However, the accuracy of the data is much lower on account of the considerable amount of impurities in the preparations. The impurities caused relatively large values of Δt , the difference between the boiling point and the temperature of condensation, as for instance 0.127° C. in the case of 2, 3-dimethylpentane. Hence the boiling points may differ from the true values for the investigated compounds by several hundredths of a degree or even more. The only exception is 2, 2-dimethyl-

TABLE 3

Data for Isomeric Heptanes

Substance	Δt	Normal boiling point	dt_s/dt_w	Boiling point of water	$(dt/dp)_{766}$
	° C	° C		° C	° C/mm
2, 2-Dimethylpentane	0.002	79.210	1.1951	100.759	
			1.1920	100.000	0.0439
			1.1875	97.755	
2, 3-Dimethylpentane	.127	89.942	1.2198	100.670	
			1.2170	100.000	. 0449
			1.2130	97.972	
3-Ethylpentane	.040	93.368	1.2179	100.726	
			1.2156	100.000	.0448
			1.2123	97.884	
3-Methylhexane	.029	91.879	1.2105	100.746	. 0445
•			1.2078	100.000	
			1.2040	97.838	
2, 2, 3-Trimethylbutane	.090	80.961	1.2174	101.041	.0448
			1.2154	100.000	
			1.2126	97.948	
n-Heptane [14]	.001	98.413	1.2179	101.034	.0448
			1.2168	100.000	
			1.2128	98.018	
				Average	0.0446

TABLE 4
Comparison of Data for Isoheptanes

Substance	Author	Normal boiling point	$(dt/dp)_{760}$
		° C	° C/mm
2, 2-Dimethylpentane	Edgar and Calingaert [8]	78.9	
	Wojciechowski	79.210	0.0439
	Brooks, Cleaton and Carter [3]	79.23	.0445
2, 3-Dimethylpentane	Edgar and Calingaert [8]	89.7	_
	Wojciechowski	89.9	.0449
3- Ethylpentane	Edgar and Calingaert [8]	93.3	_
	Wojciechowski	93.37	.0448
3-Methylhexane	Edgar and Calingaert [8]	91.8	_
	Wojciechowski	91.88	.0445
2, 2, 3-Trimethylbutane	Edgar and Calingaert [8]	80.9	
	Wojciechowski	80.96	.0448

 ${\it TABLE~5}$ Boiling Point and dt/dp Coefficient Data for 2, 2, 4-Trimethylpentane and $n\text{-}{\it Octane}$

Substance	Δt	Boiling point	dt_s/dt_w	Boiling point of water	dt/dp
	° C	° C		° C	° C/mm
2, 2, 4-Trimethylpentane [14]	0.001	99.234	1.2619	100.000	0.0465
n-Octane [22]	.008	125.658	1.2887	100.000	.0475

pentane whose Δt of 0.002° C. indicated its high degree of purity The value measured for its boiling point, 79.210° C., can differ by not more than a few thousandths of a degree from the true boiling point of 2, 2-dimethylpentane. F. W. Rose, Jr., kindly supplied a second preparation having a Δt of 0.003° C. and a boiling point of 79.208° C. The agreement of the data for the two also indicates the high purity of the preparations.

It is most probable that the preparations contain hydrocarbons isomeric with the main substance as impurities. In such a case the impurities may affect appreciably the boiling points, but can have only a very small influence on the dt_*/dt_w ratios and hence on the dt/dp coefficients. It is obvious that the preparations were good enough for precise measurements of the latter constants.

The results tabulated in Table 3 show that in the case of isoheptanes, as in the case of isohexanes, the coefficient dt/dp exhibits only small variations from the average value of 0.0446°

C/mm. The difference between the average coefficients for the hexanes and heptanes, however, is 0.0030° C/mm, an amount which is characteristic in comparison with the differences within the separate sets of isomers.

In Table 4 there are compared the author's data with those obtained by others.

(c) Isomeric octanes.—The results of measurements of the boiling points and dt/dp coefficients for n-octane and for 2, 2, 4-trimethylpentane have been reported in previous papers [22, 14]. Some data for them at their normal boiling points are given for reference in table 5. In this case the difference between the coefficients, dt/dp, for the octanes and heptanes is 0.0024° C/mm, which is again distinctive in comparison with the variations within each group.

(d) Isomeric nonanes.—Measurements of boiling points and dt/dp coefficients of n-nonane and 2, 5-dimethylheptane were made. There were used two samples of n-nonane of different purities, which permitted the calculation of the

TABLE 6

Data for Isomeric Nonanes

Substance	Δt	Normal boiling point	dt_s/dt_w	Boiling point of water	$(dt/dp)_{760}$
	°C	° C		° C	° C/mm
n-Nonane	0.016	150.81a	1.3515	100.924	0.0497
			1.3498	100.000	
			1.3460	97.902	
2, 5-Dimethylheptane	. 033	135.208	1.3276	100.623	.0489
			1.3265	100.000	
			1.3238	98.445	
2, 6-Dimethylheptane	.024	135.21	1.329	100.000	.0490
2-Methyloctane	.044	143.26	1.342	100.000	.0495
3-Methyloctane	.028	144.18	1.346	100.000	.0496
4-Methyloctane	.013	142.49	1.334	100.000	.0492
•				Average	0.0493

^{*} Extrapolated value.

boiling point of pure *n*-nonane by extrapolation. The 2, 5-dimethylheptane was supplied by J. D. White, who has reported its boiling point [21].

The data for the isomeric nonanes are given in table 6. The values for 2, 6-dimethylheptane, 2-methyloctane, 3-methyloctane and 4-methyloctane are those obtained by E. R. Smith.⁴

Although the preparations of n-nonane were not of the highest purity, since their purities might be classified on Swietoslawski's scale by the fourth and third degrees, it was possible to extrapolate the boiling point in the way formerly reported [25]. The value of 150.81° C. thus obtained for the boiling point of *n*-nonane is uncertain in the second decimal place. This value differs considerably from that of 150.6° C. reported by Edgar and Calingaert [8]. The uncertainty in the boiling point of 2, 5-dimethylheptane, 135.21°, is probably a few hundredths of a degree. The dt/dp coefficients are probably slightly affected by impurities, but not so much as to cause the difference of 0.0008 between the values of this constant for both isononanes investigated. This difference is probably due to some constitutional influence. Nevertheless, an approximately characteristic value of the dt/dp coefficient, as in the series of isomeric hexanes, heptanes and octanes, is again observed, the difference of 0.0023° C/mm between the average values for the nonanes and octanes being characteristic in comparison with the individual variations. The difference between

⁴ The data are reported in a paper by J. D. White and A. R. Glasgow, Jr. [19].

the smallest value, 0.0489, for the nonanes and the larger value, 0.0475, for the two octanes is 0.0014, which is sufficiently greater than differences within each set to be distinctive.

(e) Isomeric xylenes.—Measurements were made of boiling points and dt/dp coefficients of the three isomeric xylenes. The preparation and purification of these compounds have been described by J. D. White and F. W. Rose, Jr. [20]. The substances used in the present work were of the fourth and the third degree of purity. The data obtained for the boiling points were corrected for the influence of impurities.

Table 7 contains the data for the three xylenes.

Since the boiling points have been extrapolated to minimize the effect of impurities in the preparations, it is believed that the boiling points reported do not differ by more than one or two hundredths of a degree from the true boiling points of these xylenes. The boiling point of m-xylene is considered especially reliable since the preparation used for measurements was of the fourth degree of purity. The coefficient dt/dp, in the case of the xylenes has a fairly characteristic value. The data for m- and p-xylene are the same and equal to 0.0491, while that for o-xylene is somewhat higher. It may be worth noting that the average value for the xylenes, 0.0493, is greater than that for toluene, 0.0463, by about the same amount as appears to be more or less characteristic of the difference between adjacent sets of aliphatic isomers..

 ${\bf TABLE~7}$ Data for dt_s/dt_w and dt/dp of the Xylenes

Substance	Δt	Boiling point	Boiling point corrected	dt_s/dt_w	Boiling point of water	$(dt/dp)_{760}$
	° C	° C	° C		° C	° C/mm
o-Xylene	0.034	144.466	144.50	1.3510	101.001	0.0497
				1.3499	100.000	
				1.3483	98.503	
m-Xylene	.009	139.091	139.10	1.3344	100.633	.0491
		İ		1.3331	100.000	
				1.3304	98.821	
				1.3267	96.757	
p-Xylene	.036	138.402	138.44	1.3349	101.979	.0491
				1.3328	100.806	
				1.3316	100.000	
				1.3297	98.793	
					Average	0.0493

TABLE 8

Comparison of Data for the Xylenes

	o-Xylene		m-Xyle	ene	p-Xylene	
Observer	Normal boiling point	$(dt/dp)_{760}$	Boiling point	$(dt/dp)_{760}$	Normal boiling point	$(dt/dp)_{760}$
Mathews [13] Int. Crit. Tables [10]	° C 142.6 to 142.8	° C/mm	° C 139.3 to 139.4	° C/mm	° C 138.4	° C/mm
Huffman, Parks and Daniels [9] White and Rose [20]	144.5 144.4		139.0 139.15		138.4	
Timmermans and Martin [17] Timmermans and Henaut-Roland	111.1		100.10			0.041 to 0.042
[16]			139.30	0.049		
Wojciechowski	144.50	0.0497	139.10	.0491	138.44	0.0491

Table 8 contains a comparison of the author's data with some reported in the literature.

In Table 9 are tabulated the values for boiling points and dt/dp coefficients discussed in this paper.

An analysis of the dt/dp coefficients in the series of isomeric hydrocarbons, as listed in Table 9, leads to the conclusion that the rearrangement of a molecule results in variations of the value of the coefficient, dt/dp, which are minor in comparison with the rather characteristic difference observed between one group and the next higher group. Small deviations from the values obtained for corresponding normal compounds are probably caused by constitutional influences. This conclusion is true also in the case of the

three xylenes, which are representative of the aromatic hydrocarbons.

The results of this work confirm further the approximate additivity of the coefficient dt/dp, postulated in previous publications [22, 23]. From these results further consequences can be derived. It is well known that Trouton's rule [18] is not generally valid. The discrepancies for different compounds are so large that it is difficult to claim constancy of the ratio, L_m/T , of the molar latent heat of vaporization to the absolute temperature, even as a first approximation. The results here reported show that this ratio has a nearly constant value in the case of structural isomers. Indeed, in the equation

$$\frac{L_m}{T} = \frac{V_v - V_1}{dt/dp}$$

the right-hand member has the same value for isomeric compounds, since the value of their dt/dp coefficients is so nearly the same, and the molar volume of vapor and that of liquids are also very nearly the same for all isomers. Hence it may be postulated that the entropy of vaporization of structural isomers has a substantially constant value.

5. Summary

Normal boiling points and rates of change of boiling point with pressure of isomeric hexanes, heptanes, octanes, nonanes, and xylenes, and the freezing points of some isomeric hexanes were determined. The data are discussed and tabulated with values from other investigations.

The data confirm the rule postulated in a previous paper that the coefficient dt/dp is approximately an additive property, and show that the rearrangement of a molecule does not greatly affect the magnitude of the dt/dp coefficient. From this latter fact, the entropies of vaporization of structural isomers have substantially the same value.

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TABLE 9 Summary of Boiling Points and dt/dp Coefficients

Substance	Normal Boiling point	$(dt/dp)_{760}$
	° C	° C/mm
	Isomeric hexanes	
n-Hexane	68.733	0.0419
2-Methylpentane	60.26_{7}	.0414
3-Methylpentane	63.25_{6}	.0418
2, 2-Dimethylbutane	49.804	.0411
2, 3-Dimethylbutane	58.02_{2}	.0417
	Isomeric heptanes	
n-Heptane	98.413	.0448
2, 2-Dimethylpentane	79.21	.0439
2, 3-Dimethylpentane	89.9	. 0449
3-Ethylpentane	93.3_{7}	.0448
3-Methylhexane	91.8_{8}	.0445
2, 2, 3-Trimethylbutane	80.9_{6}	.0448
	Octane	
n-Octane	125.65_{8}	.0475
2, 2, 4-Trimethylpentane	99.23_{4}	.0465
	Isomeric nonanes	
n-Nonane	$150.8_{\rm I}$.0497
2-Methyloctane	143.2_{6}	.0495
3-Methyloctane	144.18	.0495
4-Methyloctane	142.49	.0492
2, 5-Dimethylheptane	135.2_{I}	.0489
2, 6-Dimethylheptane	$135.2_{\rm I}$. 0490
	Isomeric xylenes	
o-Xylene	144.50	.0497
m-Xylene	139.10	.0491
p-Xylene	138.44	.0491

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